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# PRELIMINARY BENEFICIATION OF A ZIRCON AND TANTALOCOLUMBITE ORE FROM THOR LAKE, N.W.T.

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# PRELIMINARY BENEFICIATION OF A ZIRCON AND TANTALOCOLUMBITE ORE FROM THOR LAKE, N.W.T.

by

M. Raicevic

## ABSTRACT

A sample of ore containing 0.4% $Nb_2O_5$ as tantalocolumbite and 3.4% $ZrO_2$ as zircon was treated to produce a high grade tantalum concentrate.

Laboratory procedures consisting of grinding, low and high intensity magnetic separtions, tabling, flotation and their combinations, were employed with the objective of producing a suitable tantalum-niobium concentrate.

The results showed that combinations of these methods produced concentrates of low niobium (tantalum) grades with relatively low recoveries. Zircon present in the ore was concentrated with the tantalocolumbite. The best final concentrate applying tabling and flotation (Procedure III Test TC-6) was as follows:

| Product | % Weight | Assay % | | Distribution % | |
|---|---|---|---|---|---|
| | | $Nb_2O_5$ | $ZrO_2$ | $Nb_2O_5$ | $ZrO_2$ |
| Cl. table conc | 7.5 | 1.76 | 24.9 | 32.7 | 74.4 |
| Slime desk conc | 1.7 | 0.96 | 6.76 | 4.0 | 4.6 |
| Flotation conc | 3.9 | 0.37 | 2.08 | 3.6 | 3.2 |
| Final (comb.) conc. | 13.1 | 1.24 | 15.7 | 40.3 | 82.2 |

The results also show that flotation had no particular effect on improvement of grades and recoveries in the final concentrate.

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# INTRODUCTION

Placer Development Ltd., Vancouver, requested that CANMET undertake beneficiation tests on a tantalum ore sample from Thor Lake district N.W.T. to produce a tantalum concentrate of the highest possible tantalum grade with maximum recovery. The work was carried out on a full cost recovery arrangement.

## ORE SAMPLE

The ore sample received was approximately 25 kg of drill core minus 1 inch in size. This sample was then crushed in stages to minus 10 mesh using a cone crusher. The sample was then split into 1 kg lots with a carousel type splitter.

Details of a mineralogy study of this ore sample are given in a report by Campbell (1). This study revealed that an important constituent of this ore sample was the mineral zircon ($ZrSiO_4$) assaying 3.4% $ZrO_2$. The major constituent of economic interest was tantalum which is mineralogically combined with niobium in a mineral called columbite or by new terminology tantalocolumbite $(FeMn)(NbTa)_2O_6$, both having equal contents in the tantalocolumbite (1). The niobium (and thus tantalum) content averaged slightly over 0.4% $Nb_2O_5$.

The tantalocolumbite, which is present in the ore in a form of elongated needle-shaped crystals averaging 20 µm long and 2 µm wide, was finely dissiminated throughout the ore and closely intergrown with other metallic and non-metallic minerals (1).

Besides the above-mentioned minerals, the others present in this ore were identified microscopically as: feldspar, albite, quartz, nephaline, soda-amphibole, biotite, green muscovite, monazite, magnetite (about 8% by weight), bastnalsite, ilmenite, rutile, pyrite, chalcopyrite and some pyrrhotite (1).

# BENEFICIATION STUDIES

## PROCEDURES TESTED

Since magnetite constitutes approximately 8% of this ore and contained relatively little niobium it was removed first by low intensity wet

magnetic separation before any other method was applied. Tabling, high intensity magnetic separation and flotation were applied at different grinds and conditions. Combinations of the methods employed will be described and illustrated separately, and detailed results tabulated from each flowsheet.

Procedure I: 200 mesh grind, low intensity magnetic separation, sizing, tabling and high intensity magnetic separation (Test TC-1).

Test TC-1

In this test 2000 gm of ore was stage ground to minus 200 mesh in a procelain mill with 1 inch steel balls. After low intensity magnetic separation to remove the magnetite the ore was then sized on a 500 mesh sieve. The +500 and -500 mesh fractions were tabled on Diester sand and slime decks respectively. The sand deck tails and concentrate were both treated by high intensity wet magnetic separation at 22,000 gauss to further recover and concentrate niobium. Even though the Sala High Gradient Magnetic Separator was at its maximum operating efficiency, the niobium recovery was low. Over 20% of the niobium was lost in the non-magnetics. In this test 78.7% of the niobium assaying 0.60% $Nb_2O_5$ was recovered in 52.1% weight of the ore.

Table 1 - Results of Test TC-1

| Product | % Wt | % $Nb_2O_5$ | % Distribution |
|---|---|---|---|
| -500 M slime deck table conc | 0.5 | 1.05 | 1.3 |
| -500 M H.I. mags | 20.1 | 0.64 | 31.9 |
| +500 M H.I. mags (fr. table conc) | 0.6 | 1.22 | 1.8 |
| +500 M H.I. non-mags (fr. table conc) | 0.4 | 0.45 | 0.5 |
| +500 M H.I. mags (fr. table tail) | 30.5 | 0.57 | 43.2 |
| Combined Nb conc | 52.1 | 0.60 | 78.7 |
| | | | |
| L.I. mags | 7.1 | 0.79 | 1.4 |
| -500 M H.I. non-mags | 23.3 | 0.10 | 15.6 |
| +500 M H.I. non-mags | 17.5 | 0.45 | 4.3 |
| | 47.9 | 0.20 | 21.3 |
| Assay head .41 Calcd head .403 | 100.0 | 0.403 | 100.0 |

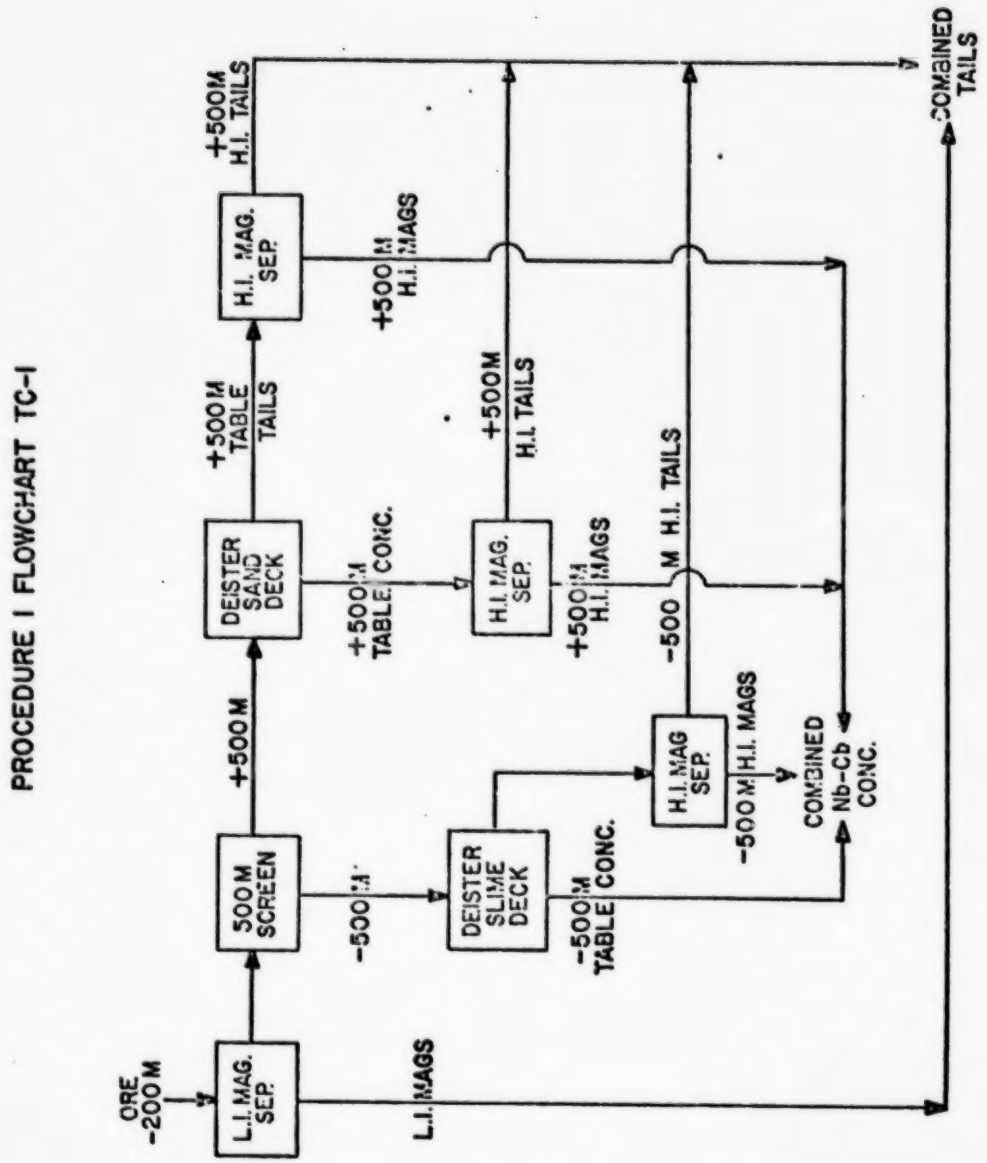



Fig.1 - Procedure I Flowchart TC-1

Procedure II: 325 mesh grind, low intensity magnetic separation, tabling and flotation of table concentrate (Test TC-5).

Test TC-5

For this test 1000 gm of ore was ground to 95% -325 M in a porcelain mill with steel balls for 1 hour. The magnetite was removed by low intensity magnetic separation and the non-magnetics were then tabled on a slime deck. The table concentrate which was approximately 13% wt of the total ore was then treated by flotation. With preliminary testing it was found that the ore had acid consumers within, thus making it difficult to maintain a low pH for flotation. The hope was that the acid consumers would be rejected in the table tails; which did happen to some extent. The pH did rise from 3.5 to 4.5 during the float. This was good compared to previous tests where the pH could not be maintained at all.

Flotation Conditions

- pH 3 - 5 using $H_2SO_4$ approximately 4 lb/ton in the rougher stage and 1.5 lb/ton in the cleaner stage.
- anionic collector was alkyl hydroxamic acid (HOE F 2773) at a rate of 4 lb/ton in rougher flotation and 1 lb/ton in cleaner stage.
- depressant was sodium silico fluoride at a rate of 0.3 lb/ton in the rougher flotation and 0.15 lb/ton in the cleaning stage.

PROCEDURE II FLOWCHART TC-5

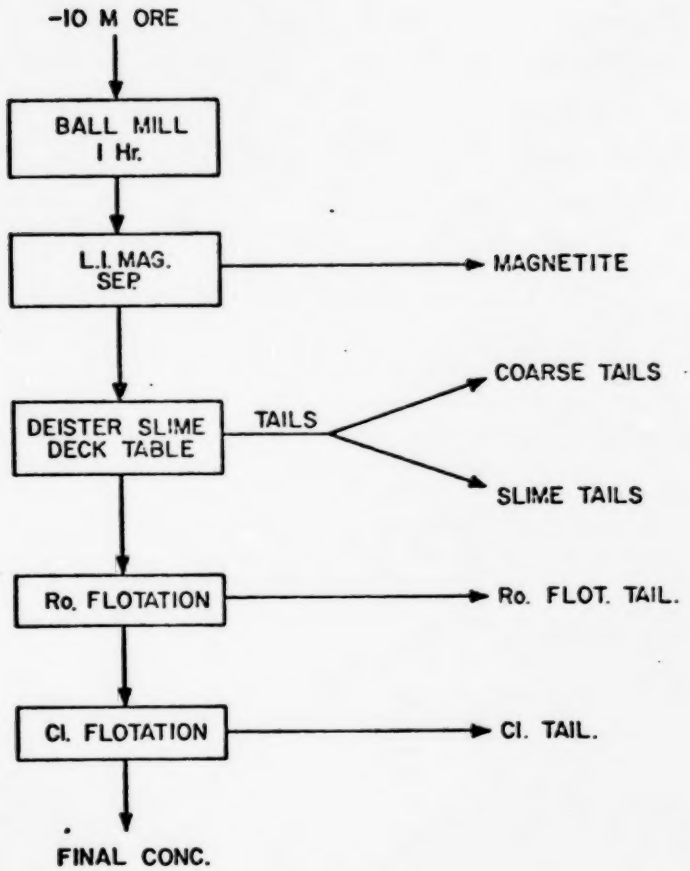



Fig. 2 - Procedure II Flowchart TC-5

Table 2 - Results of Test TC-5

| | Product | % Wt | % $Nb_2O_5$ | | % Distribution | |
|---|---|---|---|---|---|---|
| | | | XRF | Bondar | XRF | Bondar |
| 1. | Final flot conc | 1.83 | 2.19 | 0.83 | 9.9 | 3.7 |
| 2. | Cl flot tail | 3.02 | 0.90 | 0.95 | 6.7 | 7.0 |
| 3. | Ro flot tail | 7.90 | 0.52 | 0.59 | 10.1 | 11.4 |
| | Table conc (1+2+3) cald) | 12.75 | 0.85 | 0.71* | 26.7 | 22.1 |
| 4. | Coarse table tails | 67.90 | 0.36 | 0.39 | 60.1 | 64.7 |
| 5. | Slime table tail | 11.88 | 0.39 | 0.39 | 11.4 | 11.4 |
| 6. | Magnetite conc | 7.47 | 0.10 | 0.10 | 1.8 | 1.8 |
| | Final tails (4+5+6) | 87.25 | 0.34 | 0.365 | 73.3 | 77.9 |
| | Calculated heads | 100.00 | .407 | .409 | 100.0 | 100.0 |

*The table concentrate was sent to Bondar for assay. The result was 0.81% $Nb_2O_5$, somewhat higher than the calculated assay. Therefore it would seem that Bondar & Clegg's assay of one of the three products is low, most likely the final flotation concentrate for which, it seems, our assay is too high. The program for the XRF assays, performed by our laboratory, was developed by using the first set of assays obtained from test TC-1 samples which were assayed by Bondar & Clegg. These became our standards and our upper limit for % $Nb_2O_5$ was approximately 1.2%. Regardless of the concentrate assay the recovery is low.

By sizing the table tails into sands (+10 mu) and slimes (-10 mu) it was demonstrated that the niobium cannot be recovered in the slimes and that it was still locked in the coarser fraction of the tails. The tantalocolumbite mineral is so finely dessiminated throughout the ore that it is difficult to distinguish it with the microscope in any of the products produced.

Procedure III: 100 mesh grind, low intensity magnetic separation, tabling, regrinding, retabling and flotation (Test TC-6).

Test TC-6

The object of this test was to have a coarser grind -100 M and try to upgrade by tabling, retabling, superpanning and flotation. By superpanning the cleaner table concentrate it was thought that the niobium mineral could be isolated but such was not the case.

Table 3 - Results of Test TC-6

| Products | % Weight | % $Nb_2O_5$ | % Distribution | % $ZrO_2$ | % Distribution |
|---|---|---|---|---|---|
| 1. Superpanner conc | 1.2 | 1.64 | 4.9 | 31.0 | 14.8 |
| 2. Superpanner tail | 6.3 | 1.78 | 27.8 | 23.7 | 59.6 |
| (1+2) cleaner table conc | 7.5 | 1.76 | 32.7 | 24.9 | 74.4 |
| 3. Slime deck conc | 1.7 | 0.96 | 4.0 | 6.76 | 4.6 |
| 4. Flotation conc | 3.9 | 0.37 | 3.6 | 2.08 | 3.2 |
| (1+2+3+4) final conc | 13.1 | 1.24 | 40.3 | 15.7 | 82.2 |
| 5. Magnetite conc | 10.0 | 0.13 | 3.2 | 1.04 | 4.2 |
| 6. Fine rotable tail | 23.3 | 0.36 | 20.8 | 0.07 | 0.6 |
| 7. Flotation tails | 43.5 | 0.22 | 23.8 | 0.27 | 4.7 |
| 8. Slime deck table tails | 10.1 | 0.47 | 11.9 | 2.06 | 8.3 |
| (5+6+7+8) Final tails | 86.9 | 0.28 | 59.3 | 0.51 | 17.8 |
| Calculated heads | 100.0 | 0.403 | 100.0 | 2.5 | 100.0 |
| Assayed head | | 0.41 | | 3.4 | |

PROCEDURE III FLOWCHART TC-6

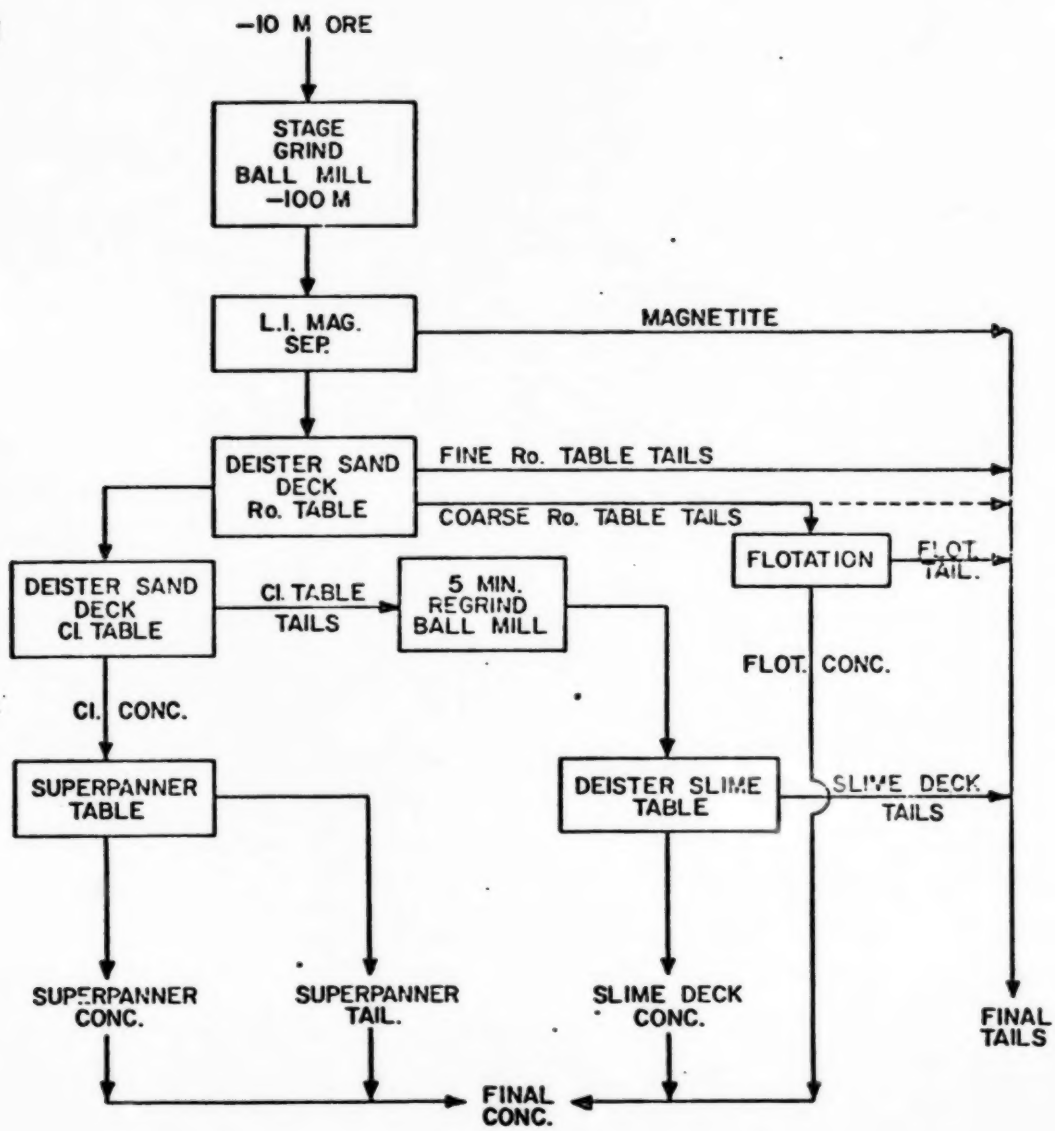



Fig. 3 - Procedure III Flowchart TC-6

This is the only test where $ZrO_2$ was assayed. As can be seen from this test it is the zirconium mineral that is being concentrated by gravity and flotation while flotation had little or no effect on the concentration of the tantalocolumbite. For the flotation of the rougher table tail sands, 2 lb/ton of HOE 2773 (alkyl hydroxamic acid) and 6 lb/ton $H_2SO_4$ were used at a pH of 4.5.

Procedure IV: 200 mesh grind, low intensity magnetic separation, tabling and flotation under various conditions (Test TC-7).

Test TC-7

The object of this test was to try different flotation reagents to recover the niobium. The ore was stage ground to -200 M and the magnetite removed. The remainder was then tabled on a Deister slime deck and the -5 micron slimes removed from the table tails. The table tails were then split into 4 fractions and subjected to different flotation procedures. None of the flotation tests provided satisfactory tailings or concentrates.

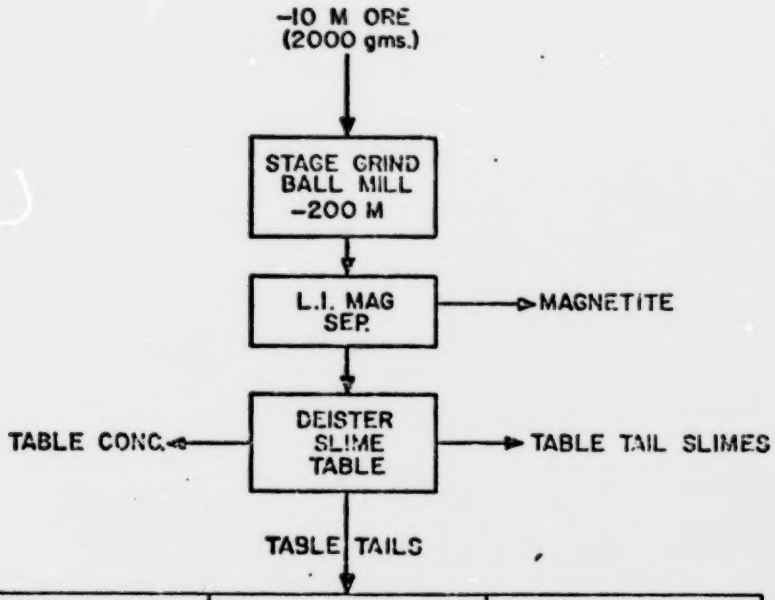
Of the four flotation procedures tried in this test only one reagent showed any selectivity and that was the hydroxamic and HOE 2773 in test TC-7 no. 2. This test produced the lowest flotation tails, 0.24% $Nb_2O_5$ and the highest concentrate approximately .65% $Nb_2O_5$ in the lowest % weight of ore. However the % recovery of $Nb_2O_5$ by flotation was only 36%.

Procecure V

Test TC-2, TC-3 and TC-4

Stage grind to -325 mesh in ball mill (porcelain mill, with steel balls); remove magnetite by low intensity magnetic separation then flotation with various known reagents and conditions (Test TC-2, TC-3 and TC-4).

## PROCEDURE IV FLOWCHART TC-7




| TEST 1 | TEST 2 | TEST 3 | TEST 4 |
|---|---|---|---|
| CONDITIONS: | CONDITIONS: | CONDITIONS: | CONDITIONS: |
| ARMAK - Doumeen TDO<br>Fatty acid 1.7 lbs/ton<br>$H_2SO_4$ 13 lbs/ton<br>ph 2 | Hydroxamic acid HOE2773<br>.2 lbs/ton ro. Flot<br>.15 lbs/ton Scav. Flot.<br>$H_2SO_4$ 8.5 lbs/ton Ro. Flot.<br>1.5 lbs/ton Scav. Flot.<br>ph 5-6 | Neutral ph 8.4<br>Oelic acid .1 lbs/ton<br>Sodium Silicate .3 lbs/ton | Leach in 10% HCl ? 4 hrs wash + filter<br>Neutralize with NaOH to ph 7<br>Condition to ph 2 with 1.5 lbs/ton $H_2SO_4$ ph 2<br>ARMAK-DUOMEEN TDO 1.5 lb/ton |

**Fig. 4 - Procedure IV Flowchart TC-7**

Table 4 - Results of Test TC-7

| Product | % Wt | % $Nb_2O_5$ | % Distribution |
|---|---|---|---|
| Test TC-7 no. 1 | | | |
| Table conc | 6.8 | 0.96 | 15.9 |
| Final flot conc | 3.4 | 0.52 | 4.3 |
| 1st cl tail | 4.4 | 0.42 | 4.5 |
| 2nd cl tail | 12.9 | 0.42 | 13.2 |
| Total conc | 27.5 | 0.57 | 37.9 |
| Flot. tails | 59.9 | 0.31 | 56.0 |
| L.I. mags | 7.7 | 0.07 | 1.5 |
| Table tail simes | 4.9 | 0.39 | 4.6 |
| Total tails | 72.5 | 0.35 | 62.1 |
| Calculated heads | 100.0 | 0.41 | 100.0 |
| Test TC-7 no. 2 | | | |
| Table conc | 6.8 | 0.96 | 17.8 |
| Ro flot conc | 8.1 | 0.63 | 13.9 |
| Scav flot conc | 12.0 | 0.67 | 21.9 |
| Total conc | 26.9 | 0.73 | 53.6 |
| Flot tail | 60.5 | 0.24 | 39.5 |
| L.I. mags | 7.7 | 0.07 | 1.7 |
| Table tail slimes | 4.9 | 0.39 | 5.2 |
| Total tails | 73.1 | 0.23 | 46.4 |
| Calculated heads | 100.0 | 0.37 | 100.0 |
| Test TC-7 no. 3 | | | |
| Table conc | 6.8 | .96 | 17.8 |
| Ro flot conc | 10.6 | .29 | 8.4 |
| Total tails | 17.4 | .55 | 26.2 |
| Ro flot tail | 70.0 | .35 | 66.9 |
| L.I. mag | 7.7 | .07 | 1.7 |
| Table tail slimes | 4.9 | .39 | 5.2 |
| Total tails | 82.6 | .33 | 73.8 |
| Calculated heads | 100.0 | .37 | 100.0 |
| Test TC-7 no. 4 | | | |
| Table conc | 6.8 | .96 | 16.1 |
| Flot conc | 27.5 | .57 | 38.6 |
| Total conc | 34.3 | .65 | 54.7 |
| Flot tail | 53.1 | .30 | 39.1 |
| L.I. mag | 7.7 | .07 | 1.5 |
| Table tail slimes | 4.9 | .39 | 4.7 |
| Total tails | 65.7 | .28 | 45.3 |
| Calculated heads | 100.0 | .41 | 100.0 |

Table 5 - Results of Tests TC-2, TC-3 and TC-4

| Test No. | Flotation Reagents and Conditions | Product | % Weight | % $Nb_2O_5$ | % Distribution |
|---|---|---|---|---|---|
| TC-2 | Potassium octylhydroxamate | Flot conc | 27.6 | 0.48 | 35.9 |
| | .2 lb/ton | Flot tail | 64.4 | 0.36 | 62.6 |
| | $H_2SO_4$ - pH 6 - 14 lb/ton | L.I. mag | 8.0 | 0.07 | 1.5 |
| | Pine oil - .01 lb/ton | Calculated heads | 100.0 | 0.37 | 100.0 |
| TC-3 | Hydroxamic acid | Flot conc | 16.3 | 0.56 | 22.9 |
| | HOE 2773 - 5.5 lb/ton | Flot tail | 77.0 | 0.39 | 75.4 |
| | $H_2SO_4$ - pH 6 - 10 lb/ton | L.I. mag | 6.7 | 0.10 | 1.7 |
| | | Calculated heads | 100.0 | 0.40 | 100.0 |
| TC-4 | Hydroxamic acid | | | | |
| | HOE 2773 - 5.5 lb/ton | Flot conc | 12.2 | 0.53 | 16.5 |
| | H2SO4 - pH 6 - 10 lb/ton | Flot tail | 81.0 | 0.40 | 81.8 |
| | Sodium-silica fluoride | L.I. mag | 6.8 | 0.10 | 1.7 |
| | 5 lbs/ton | Calculated heads | 100.0 | 0.40 | 100.0 |

Procedure VI: 200 mesh grind, low intensity magnetic separation, with (TC-8) and without (TC-9) HCl leaching and flotation.

Test TC-8, TC-9

Procedures for TC-8 included stage grinding the ore to -200 mesh in porcelain mill with porcelain balls; low intensity magnetic separation, desliming during magnetic separation, HCl leaching and then flotation. General procedure for test TC-9 was the same as TC-8 except there was no HCl leach and the reagents were different for the flotation. By using the pebble mill in both tests it was thought that flotation may be improved.

PROCEDURE VI FLOWCHART TC-8

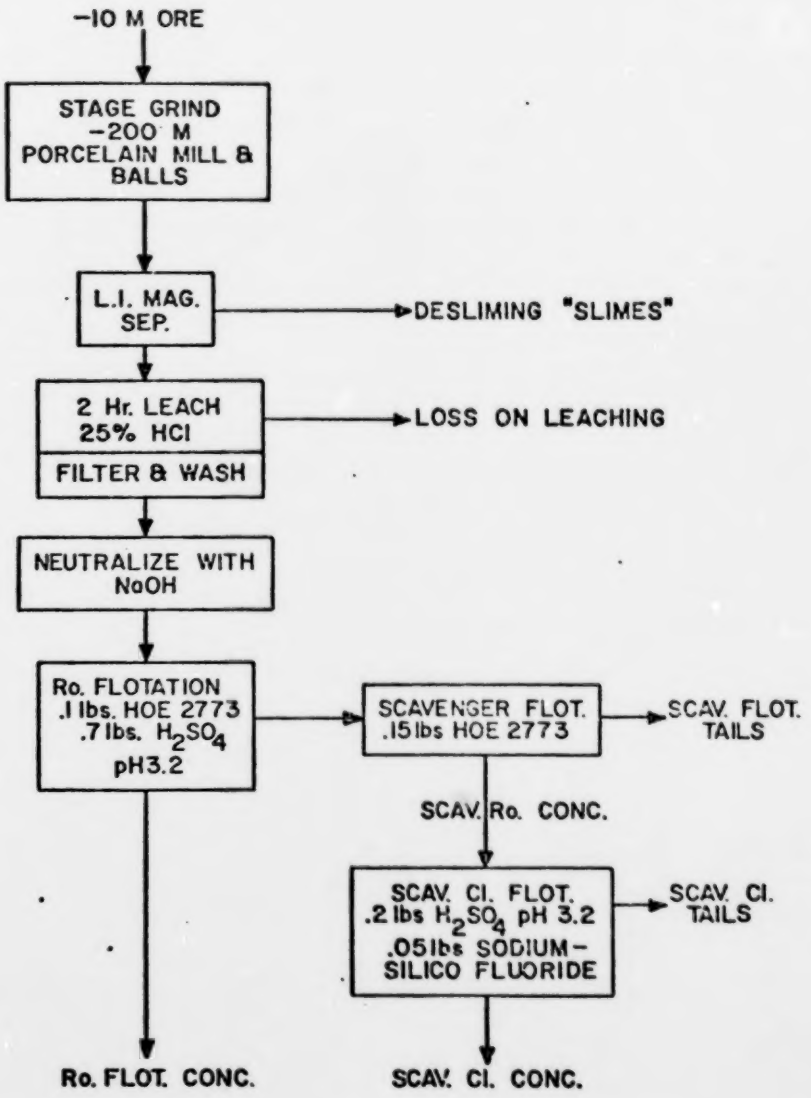



Fig. 5 - Procedure VI Flowchart TC-8

PROCEDURE VI FLOWCHART TC-9

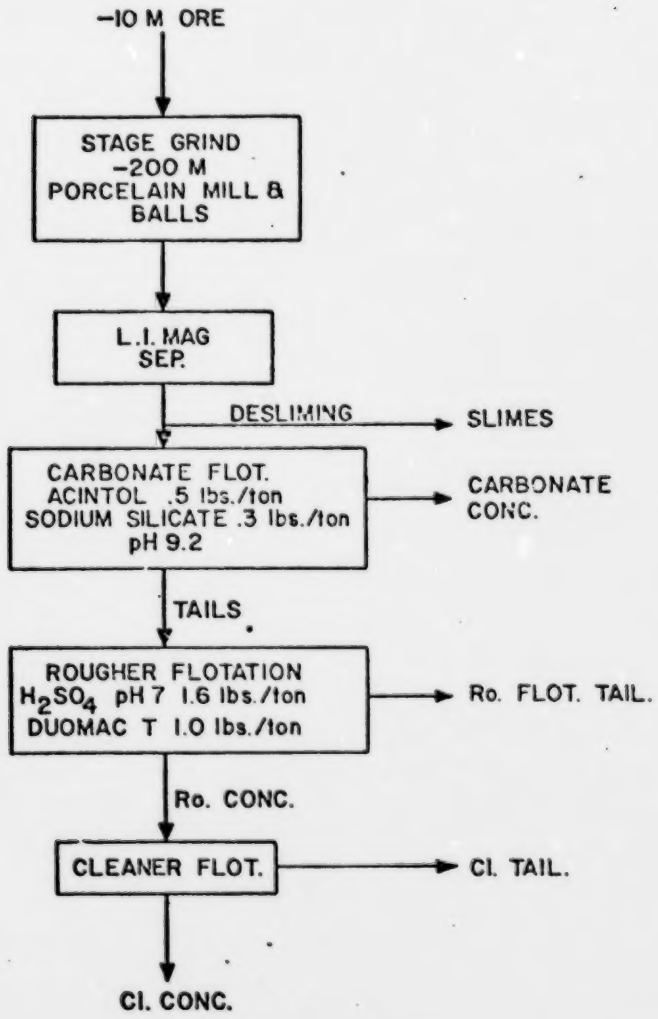



Fig. 6 - Procedure VI Flowchart TC-9

Table 6 - Results of Tests TC-8 and TC-9

| Product | % Wt | % $Nb_2O_5$ | % Distribution |
|---|---|---|---|
| Test TC-8 | | | |
| Ro flot conc | 1.9 | 0.56 | 2.4 |
| Scav cl conc | 1.9 | 0.83 | 3.6 |
| Scav cl tail | 5.8 | 0.78 | 10.3 |
| | 9.6 | 0.74 | 16.3 |
| Scav flot tails | 66.8 | 0.50* | 76.3 |
| Slimes | 6.3 | 0.42 | 6.1 |
| L.I. mags | 8.0 | 0.07 | 1.3 |
| Loss on HCl leach | 9.3 | --- | --- |
| Total tails | 90.4 | 0.37 | 83.7 |
| Calculated heads | 100.0 | 0.44* | 100.0 |
| Test TC-9 | | | |
| Carbonate conc | 7.4 | 0.35 | 6.0 |
| Cleaner conc | 14.1 | 0.39 | 12.8 |
| Cleaner tail | 9.3 | 0.26 | 5.6 |
| Rougher tail | 55.3 | 0.53* | 68.3 |
| L.I. mags | 7.8 | 0.07 | 1.3 |
| Slimes | 6.1 | 0.42 | 6.0 |
| Calculated heads | 100.0 | 0.43* | 100.0 |

*Assays were higher than expected.

In test TC-8, HCl leaching was introduced to remove the acid consumers that are in the ore so that the pH could be maintained throughout flotation. The recommended pH for the anionic collector HOEF 2773 is between pH 3-5. In this test the pH of 3.2 remained constant throughout the entire flotation procedure. In the scavenger operation twice as much collector HOE 2773 was added and the results were better. Also what might have improved the flot in the scavenger was the length of conditioning time. Conditioning time in the rougher flotation stage was 5 minutes whereas the scavenger stage had only 1 minute. It would seem that the effectiveness of the reagent deteriorates

with time. Thus shorter or almost nil conditioning might possibly improve the grade or recovery, but this point was not further investigated. When the results of this test TC-8 are compared to the results from others using HOE 2773 such as TC-3 and TC-4, it can be seen that TC-8 had the highest grade in the lowest % weight but somewhat lower recovery.

In test TC-9 a carbonate flotation procedure using acintol and sodium silicate to remove the acid consumers was applied. However, during the Nb float the pH still climbed from pH 6 to 7.5 throughout the test. Another type of collector, Duomac T was tried but with no encouraging results.

In both of these tests the calculated $Nb_2O_5$ assay is higher than the assayed head, indicating that there is a high assay and looking at the results it would appear that the anomaly could be in the flotation tails which are reported to be 0.50% and 0.53% $Nb_2O_5$.

Procedure VII: 200 mesh stage grind, low intensity magnetic separation and flotation (Test TC-10).

Test TC-10

A 1000 gm charge of ore was stage ground to -200 mesh in a porcelain mill with steel balls. The magnetite was then removed by low intensity magnetic separation. The remainder was then subjected to flotation using 1.0 lb/ton sodium oleate and 3 lb/ton sulphuric acid at a pH of 5.5. By the end of the flotation test, the pH was back up to 7.5.

Table 7 - Results of Test TC-10

| Product | % Wt | % $Nb_2O_5$ | % Distribution |
|---|---|---|---|
| Ro flot conc | 53.7 | 0.51 | 71.7 |
| Ro flot tail | 38.8 | 0.28 | 27.9 |
| L.I. mags | 7.5 | 0.07 | 1.4 |
| Calculated heads | 100.0 | 0.39 | 100.0 |

## CONCLUSION

The tantalocolumbite mineral is finely dissiminated throughout the ore and closely intergrown with other minerals. Because of its apparent brittleness and crystal formation (elogated needle-shaped crystals) (1), it is difficult to grind to liberation without incurring high losses in the fines. As a result it has been found that the tantalocolumbite could not be satisfactorily concentrated by tabling, magnetic separation, flotation and their combinations, but a fairly good zircon concentration was achieved.

## REFERENCES

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